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(54) PROVIDING A LAYER ON A SUBSTRATE

- (71) We, PHILIPS ELECTRONIC AND ASSOCIATED INDUSTRIES LIMITED, of Abacus House, 33 Gutter Lane, London, EC2V 8AH, a British Company, do hereby declare the invention, which was communicated from N.V. Philips' Gloeilampenfabrieken, a limited liability company, organized and established under the laws of the kingdom of the Netherlands, of Emmasingel 29, Eindhoven, Holland, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- The present invention relates to a method of providing a layer of solid material on a flat substrate surface, in which a drop or drops of liquid material from which the solid material is formed is allowed to fall onto the substrate while the substrate is rotated about an axis passing through the substrate.
- The invention is very suitable for the manufacture of slices and layers of a semiconductor material, especially silicon, of large area for use in the manufacture of semiconductor devices, in particular solar cells.
- The use of solar energy as an alternative to the other, usual sources of energy is at present still restricted, in particular due to the high cost-price of the devices for receiving and converting said energy. Particularly for the manufacture of solar cells, attempts have been made to develop production methods with which the cost-price of the cells can be reduced considerably compared with production by the current methods. For example, in the manufacture of silicon solar cells, the method used for the manufacture of monocrystalline plates from which the cells are manufactured is still complicated. First, monocrystalline silicon rods are manufactured which are then divided into wafers by sawing, and the wafers are subsequently polished so as to remove the rough surfaces by the sawing operation. In each of these long and expensive operations a part of the silicon is lost; moreover, losses as a result of the numerous quality checks which are inevitable in each stage of manufacture must be taken into account. The complicated character and the duration of the preparation processes, together with the high final loss percentage of silicon (about 50% by weight with respect to the polycrystalline starting material), cause the manufacture of solar cells of monocrystalline material to be rather expensive.
- It has therefore been tried to develop methods for the manufacture of solar cells manufacture of monocrystalline rods and of polycrystalline material in which the subsequent sawing operations, and possibly even certain surface treatments, may be omitted, and in which nevertheless wafers or layers provided on substrates can be obtained of which the quality of the surface and the purity are acceptable. It is known that the efficiency of solar cells of polycrystalline material in converting light energy into electrical energy can reasonably approach that of the monocrystalline solar cells if the polycrystalline material is of a reasonable quality.
- A method of manufacturing wafers of semiconductor material of a great purity for the manufacture of solar cells is described in United Kingdom Patent Specification No. 1,032,071. According to this method the semiconductor material is provided in powder form in a layer of small and uniform thickness on a flat support and is exposed to an electron bombardment. This bombardment produces a melting of the surface of the grains, which consequently flow together and solidify so as to form a polycrystalline layer of small thickness.
- Such a method permits of obtaining a layer of good purity, as described in the said patent specification. The method requires a comparatively expensive apparatus, however, and must be carried out accurately so as to obtain a readily coherent layer.

It is the object of the present invention to provide a method with which a layer can be provided in a simple manner on a substrate of a substantially large area.

5 It is known to provide a layer of solid material on a flat substrate surface by allowing a drop or drops of a liquid material from which the solid material is formed to drop onto the substrate while the substrate is rotated about an axis passing through the substrate. This known method is used, for example, in semiconductor technology for providing a layer of photolacquer on a substrate. By rotation of the substrate the photolacquer which is supplied to the substrate in drop form, is spread over the substrate surface. It has been found, however, that in practice a layer provided in this manner may be non-uniformly distributed over the substrate surface, with the result that the layer thickness in the area where the axis of rotation passes through the substrate may differ from the layer thickness nearer the periphery of the substrate. This may be the case particularly if the liquid material has a high surface tension. It is a further object of the invention is to obtain a better distribution of the material over the substrate surface.

30 According to the invention, a method of providing a layer of solid material on a flat substrate surface in which a drop or drops of a liquid material from which the solid material is formed is or are allowed to fall onto the substrate while the substrate is rotated about an axis passing through the substrate, is characterized in that the rotating movement of the substrate is combined with at least one other periodic movement which produces a change of the position of the plane of the substrate surface such that points of the substrate surface which are situated outside the point where the axis of the rotating movement passes through the substrate vary in height with respect to the latter point.

A homogeneous spreading is further promoted if the frequencies of the rotating movement and the other periodic movement are different. The frequency of the other periodic movement is preferably larger than the frequency of the rotating movement. The frequency of the rotating movement may then be restricted so as to prevent liquid from being thrown off the substrate by centrifugal force, while by the combination of the rotating movement with the more rapid other periodic movement a rapid spread of the dropped liquid material can be obtained.

60 In principle the axis of the rotating movement can remain vertical while the other periodic movement varies the position of the plane of the substrate surface relative to

said axis of rotation, for example, by using a tilting movement.

According to a preferred embodiment the axis of the rotating movement remains at right angles to the substrate surface and the other periodic movement varies the position of the axis of the rotating movement simultaneously with the position of the plane of the substrate surface. The other movement may be a tilting movement, preferably with extreme positions which are at equal angles to the intermediately located horizontal position. Even a small angle of tilt, that is, the angle between the two extreme positions, will produce a noticeable improvement of the spreading of the dropped liquid. When the angle of tilt is too large the danger exists of the occurrence of a continuously varying local thickness variation and of the flowing away of liquid from the substrate surface. Suitable values of the angle between the two extreme positions of the substrate surface lie, for example, between 3° and 10°.

In the case in which the axis of the rotating movement always remains normal to the substrate surface, the other periodic movement may be a rotating movement which varies the position of the plane of the substrate surface, the axis of the other rotating movement being inclined with respect to the normal to the substrate surface; for example, this axis may be vertical whereas the positions of the substrate surface are inclined. The angle of inclination of said axis may also be comparatively small, preferably in the case in which a partial flowing away of the dropped liquid should preferably be obtained. Suitable values of said angle lie, for example, between 1.5° and 5°.

By the combination of a rotating movement and another periodic movement which varies the position of the plane of the substrate surface, it is possible to drop the liquid material eccentrically of the axis of the rotating movement, which means that the place where the liquid material is dropped on the substrate surface is situated outside the axis of the rotating movement. As a result of this, the possibility of undesired thickening of the layer of material in the region of the axis of rotation, where the centrifugal effect is at a minimum, is further reduced. It is to be noted that in the known method which employs only a rotating movement, if the liquid material is dropped eccentrically the substrate, as a result of the centrifugal effect, may remain uncovered in the region of the axis of rotation. With the added periodic movement in accordance with the present invention, use is made of the additional effect of gravity for the flow of liquid towards the point where the axis of rotation passes through the substrate.

The distance between the axis of rotation and the average point of impact of the drop or drops of the liquid material is a determining factor in the time taken for the whole substrate to become covered with the dropped material. For optimum covering the most favourable distance will be chosen in accordance with the size of the substrate. For a circular substrate surface, the above distance may, for example, be between 1/10 and 1/3, preferably between 1/5 and 1/4, of the diameter of the substrate surface.

The method according to the invention may be used to obtain layers of various solids, for example, layers of synthetic resins, for example, by hardening of monomeric or (partially) pre-polymerized liquid starting material, thermoplastic material which can be heated to the liquid state, in solvents, in particular in volatile solvents, or soluble solid material. The method may be applied in particular to meltable material which will solidify after or during spreading over the substrate surface. The substrate may be heated to just below the melting point of the material of the layer to be formed, in such a manner that solidification occurs sufficiently slowly for the material to be able to spread while it is still liquid. In the case of a crystalline material the method according to the invention permits of obtaining layers of a substantially non-porous structure, for example, a substantially homogeneous polycrystalline structure having a smooth reflective surface. Also for the above reasons the method according to the invention has proved to be very suitable for providing layers of semiconductor material having a fairly large area. Although in general the semiconductor material is in this manner obtained in a polycrystalline form, the layers nevertheless prove to have an acceptable quality for the manufacture of cheap semiconductor devices, in particular solar cells, with a reasonable efficiency. Mechanical finishing treatments of the surface of the formed layer may be omitted. The method has even proved suitable for the formation of silicon layers of acceptable semiconductor quality, notwithstanding the very high melting-point of silicon.

The choice of the frequencies of the rotating movement and the other periodic movement may be made on the basis of experiments. Good results have been obtained, for example, with frequencies of the rotating movement between 15 and 30 rpm and frequencies of the other periodic movement between 3 and 6 oscillations per second.

As regards the volume of the liquid material supplied to the substrate, this may correspond to one or several drops of liquid material. If several drops are used for the formation of a layer on one substrate, the

frequency of the falling drops should be sufficiently high to ensure that the solidification of the liquid material occurs substantially continuously. Otherwise, the rate of crystallization for meltable crystalline material may be influenced by temperature variations of the substrate.

It has proved possible with the method according to the invention to form on a circular substrate a layer having a diameter of 100 mm and a thickness of between 10 and 400 μm .

With such layers of semiconductor material, in particular of silicon, solar cells of a reasonably large area can be cheaply manufactured. The semiconductor material in the liquid state may be doped in such a manner that the semiconductor layer which is obtained from this material may obtain a desired extrinsic conductivity type. The semiconductor layer may then be subjected to a diffusion treatment so as to form therein a surface zone, the conductivity type of which is opposite to the original type so that a $p-n$ junction of large area is obtained. The formed layer preferably remains connected to the substrate on which it is crystallized, which substrate gives the semiconductor layer mechanical rigidity.

For a silicon layer a substrate of graphite may be used, which substrate may form an ohmic contact with the silicon layer. A second contact, which preferably should be sufficiently permeable to solar rays, is provided on the surface where the surface zone of the opposite conductivity type is formed. It is alternatively possible to form a self-supporting layer which can be removed from the substrate.

Using the method according to the invention, the cost-piece of a solar cell may be considerably lower than that of cells of mono-crystalline material manufactured according to conventional methods, first of all due to the reduction of the number of operations and, furthermore, because it is possible to obtain elements of larger area. For very large areas drops may be released from a number of locations, suitably distributed above the substrate surface.

Embodiments of the invention will be described in greater detail with reference to the accompanying drawings, in which

Fig. 1 shows diagrammatically an apparatus for providing a layer of solid material on a substrate by means of a method according to a first embodiment of the invention, and

Fig. 2 shows diagrammatically a device for producing a combination of a rotating movement and another periodic movement of a flat substrate which is to be covered with a layer of solid material by means of a method according to a first embodiment of the invention.

In the apparatus shown in Fig. 1 the material from which a layer of solid material is to be obtained is provided in a crucible 1, in which the material is heated by means of heating elements 3 to a temperature which is sufficiently high to form a liquid mass 2 of the material.

A conical nozzle 4 is provided in the bottom of the crucible 1 so that the liquid material 2 can flow from the crucible in the form of drops 5. A vertically movable valve element 6, the lower end of which is shaped to fit in the nozzle 4, permits of either closing the nozzle or controlling the frequency of the drops by adjusting the valve element axially in the nozzle.

The crucible 1 preferably performs a rotating movement so as to keep the liquid mass 2 homogeneous.

The drops 5 fall onto a flat substrate 7 which is rotated about an axis AA by a turntable 8 on which the substrate 7 is mounted, the turntable 8 itself being rotated by a spindle 9 which rotates, for example, in the direction denoted by the arrow F_1 .

The mechanical connection between the turntable 8 and the spindle 9 is provided by a ball joint 10, so that the regular rotating movement thereof to produce an oscillation of the substrate on opposite sides of a substantially horizontal plane. This tilting movement is in Fig. 1 by the double arrow F_2 . The angle of tilt, that is, the angle between the two planes which correspond to the highest dead point (for example, when the substrate 7 is in the position 7a denoted in broken lines) and the lowest dead point (for example, when the substrate 7 is in the position 7b denoted in broken lines) of the tilting movement, is preferably between 3° and 10° . This movement can be produced, for example, by directing local jets of gas against the lower side of the turntable 8 at points located eccentrically of the axis AA through gas supply tubes provided for that purpose (not shown).

The spindle 9 and the crucible 1 are arranged so that there is a certain distance d between the substantially vertical axis AA about which the rotation of the substrate is carried out and the vertical path BB of the drops 5.

The apparatus furthermore comprises a heating element 11 which is preferably provided, as is shown in Fig. 1, above the edge of the substrate 7. The temperature of the substrate 7 may thus be adjusted and the duration of the crystallization thus be controlled.

Furthermore, the heating element 11, due to the delaying effect it exerts on the crystallization, permits the use of a number of successive drops for the manufacture of a single layer of solid material.

The crucible 1, the heating elements 3 and 11, the turntable 8 and the upper part of the spindle 10 are accommodated in a vessel through which a suitable gas flows. The vessel is indicated diagrammatically by the rectangle 12, omitting any reference regarding passages or flow tubes for the gas.

The means shown in Fig. 1 for moving the substrate 7, may be replaced by the device shown in Fig. 2.

The device shown in Fig. 2 comprises a vertical spindle 23 which at its lower end bears on a horizontal supporting block 21, the central part of which comprises a sleeve 22 in which the lower end of the spindle 23 is journaled so that the spindle is capable of rotating freely about a vertical axis. The spindle 23 forms an assembly with two gear wheels: a first bevel gear wheel 24 which is secured to the upper end of the spindle 23 and a second bevel gear wheel 25 which is secured to a lower part of the spindle 23. The bevel gear wheel 25 together with a bevel gear wheel 26 which is mounted on a shaft 27 of a driving motor not shown, constitutes a bevel-gear coupling for transmitting a rotating movement to the spindle 23.

A cylindrical sleeve 28 rotatably surrounds the spindle 23. At its lower end the sleeve 28 is journaled so as to be freely rotatable on a supporting collar 23a on the spindle 23. Furthermore, the sleeve 28 is provided near its lower end with a bevel gear ring 29 which meshes with a bevel wheel 30 which is secured on the shaft 31 of a driving motor not shown. The sleeve 28 has a laterally extending arm 32 with a cylindrical portion 32a which is inclined at an acute angle to the spindle 23, the respective axes CC and AA of the arm portion 32a and the spindle 23 intersecting each other in a point G which is situated above the gear wheel 24. Supported on the portion 32a of the arm 32 is a cylindrical sleeve 33 which is closed at its upper end and can rotate freely about the arm portion 32a.

The cylindrical sleeve 33 has a bevel gear wheel 34 fixed on its upper end. Also fixed on the upper end of the sleeve 33 is a spindle 35 which is coaxial with the sleeve 33.

The bevel gear wheel 24 on the spindle 23 meshes with the bevel gear wheel 34 on the sleeve 33. The spindle 35 supports the turntable 8 the upper surface of which is at right angles to the axis CC. A graphite plate 7 which forms the substrate on which the material to be crystallized is deposited, is laid on the upper surface of turntable 8. The centre of the upper surface of the plate 7 substantially coincides with the point of intersection G of the axes AA and CC, so that both upon rotation of the sleeve 28

and upon rotation of the spindle 35 the centre of the upper surface of the plate 7 substantially does not change position.

In principle the above device has three inter-related parts which can perform different rotational movements, namely

1. the spindle 23 with the gear wheels 24 and 25,
- 10 2. the sleeve 28 with the gear wheel 29 and the arm 32,
3. the sleeve 33 with the gear wheel 34, the spindle 35, the turntable 8 and the plate 7.

The movement of the turntable 8 and of the plate 7 is the result of both the rotating movement of the arm 32 by rotation of the sleeve 28 about the spindle 23, and the rolling of the gear wheel 34 around the gear wheel 24 during the rotation of the sleeve 28.

If the spindle 23 and the sleeve 28 rotate in the same direction at the same speed, there is no relative rotation between the gear wheels 24 and 34. The plate 7 then rotates exclusively about the axis AA and not about the axis CC. This varies the orientation of the plate 7 in space but all points of the plate describe circles in planes which are parallel to each other and at right angles to the axis AA, with the result that all the points of the surface to be covered remain at constant levels.

If the spindle 23 and the sleeve 28 rotate at different speeds and/or in opposite directions, relative rotation occurs between the gear wheel 34 and the gear wheel 24. This causes a rotation of the plate 7 about the axis CC and consequently an oscillating variation of the height of each of the points of the surface of the plate 7 which are to be covered, with the exception of the point G, with respect to the point where the axis CC passes through the surface of the plate 7, which in this case coincides with the point G.

The ratio of the frequency of the oscillation in height of the points, other than the point G, of the substrate surface of the plate 7 (substrate) with respect to their rotation frequency in space depends on the respective angular speeds of the gear wheels 24 and 34.

By suitably controlling the direction and speed of rotation of the gear wheel 24 with a given direction and speed of rotation of the sleeve 28 about the spindle 23, it can even be achieved that the various points of the plate 7 stop rotating in space and only perform a regular up-and-down movement. This is obtained, for example, in the case in which with equally proportioned gear wheels 24 and 34 the spindle 23 rotates

in the same direction as the sleeve 28 at a speed which is twice that of the sleeve.

As a result of the rotating movement of the normal to the substrate surface about the vertical, the direction of flow of liquid at a given point on the substrate surface will always vary, so that in the last-described case also a dropped liquid can spread over the whole substrate surface.

By way of example, the use of the apparatus shown in Fig. 1, but with the movement device shown in Fig. 2, for providing a layer of silicon on a flat substrate will now be described.

The substrate 7 of graphite is circular and has a diameter of 100 mm and a thickness of 100 μ m. It is heated on the turntable 8 to a temperature between 1390° and 1410°C. The heating element 11 is arranged at a height of 5 cm above the level of the centre of the substrate surface.

The height of the outlet of the nozzle 4 may be adjusted to between 8 and 12 cm, measured from the centre of the substrate surface. The heating element 3 heats and maintains the silicon in the crucible 1 at a temperature which is higher than the melting temperature, for example 1430°C. A constant flow of argon is passed through the vessel 12. According to a modified embodiment, argon or another protective gas at a reduced pressure of about 16 mm mercury may alternatively be used in the vessel.

The orientation of the substrate surface in space is varied by means of a rotation of the axis CC about the vertical axis AA with a frequency of 5 revolutions per second (300 rpm). These two axes, which intersect each other in the point G in the centre of the surface of the substrate 7 to be covered, form an acute angle of 3° with each other. Since the axis CC is also at right angles to the substrate surface, the substrate surface has an inclined position deviating at an angle of 3° from a horizontal position. Since the axis AA is vertical, the substrate surface, in its various positions during the said periodic movement, will always deviate by an angle of 3° from the horizontal position. The said movement of the axis CC about the vertical axis AA is obtained by rotating the sleeve 28 at said speed of 300 rpm.

In order to vary the heights of the points of the surface of the substrate situated outside the point G, the spindle 23 with the gear wheel 24 should not be rotated at the same speed (in the same direction) as the sleeve 28. Furthermore, it is desirable that the horizontal speed of movement of the place on the substrate surface on which a drop of liquid material falls should not be too large, so as to avoid the drop being thrown off the surface by centrifugal force.

In the present example a rotation about the axis CC is added such that the points of the substrate surface situated outside the point G perform rotations about the axis AA with a frequency of only 20 rpm. For this purpose the spindle 23 is rotated in the same direction as the sleeve 28 but with a frequency of 580 rpm, i.e., twice the frequency of the sleeve 28 reduced by the desired frequency of the revolutions of the points on the substrate surface.

The distance d (see Fig. 1) between the vertical path of the molten drops and the vertical axis AA is 20 mms.

The average volume of a drop 5 is 20 mm^3 . Twenty drops of molten silicon are allowed to fall on the substrate at a frequency of 3 drops per second.

In this manner a layer of polycrystalline silicon is provided over the whole upper surface of the plate 7. The layer has a uniform thickness of $50 \mu\text{m}$ and its crystallization is completed between 6 and 10 seconds after the last of the twenty drops has fallen. This layer adheres very intimately to the underlying graphite and has a very shiny appearance which is an indication of a good homogeneity and purity.

The graphite plate 7 which is covered with the layer of polycrystalline silicon may be used for the manufacture of a solar cell dependent upon the extent to which the silicon has suitably been doped. For this purpose, a suitable quantity of dopant may previously be added to the quantity of liquid material 2. The dopant may be boron. The crystalline silicon of the resulting layer then has a p -type conductivity. To a depth of $0.3 \mu\text{m}$ from the surface of the polycrystalline layer, a zone of n -conductivity is formed by diffusion of phosphorus, this zone forming a pn junction with the underlying p -type silicon. A contact with the n -type zone is provided on the surface of the said layer, while the other contact of the cell is provided on the uncovered surface of the graphite plate 7.

WHAT WE CLAIM IS:—

1. A method of providing a layer of solid material on a flat substrate surface, in which a drop or drops of a liquid material from which the solid material is formed is or are allowed to fall onto the substrate while the substrate is rotated about an axis passing through the substrate, characterized in that the rotating movement of the substrate is combined with at least one other periodic movement which produces a change of the position of the plane of the substrate surface such that points of the surface which are situated outside the point where the axis of the rotating movement passes through the substrate vary in height with respect to the latter point.

2. A method as claimed in Claim 1, characterized in that the other periodic movement has a different frequency from that of the rotating movement.

3. A method as claimed in Claim 2, characterized in that the other periodic movement has a larger frequency than the rotating movement.

4. A method as claimed in any of the Claims 1 to 3, characterized in that the axis of the rotating movement remains at right angles to the substrate surface and the other periodic movement varies the position of the axis of the rotating movement.

5. A method as claimed in any of the preceding Claims, characterized in that the other periodic movement is a tilting movement.

6. A method as claimed in Claim 5, characterized in that the maximum angle of tilt is between 3° and 10° .

7. A method as claimed in Claim 4, characterized in that the other periodic movement is a rotating movement about an axis which is inclined with respect to the normal to the substrate surface.

8. A method as claimed in Claim 7, characterized in that the angle of the inclination of the axis of the other rotating movement to the normal to the substrate surface is between 1.5° and 5° .

9. A method as claimed in any of the preceding Claims, characterized in that the place where the liquid material is dropped on the substrate surface is situated outside the axis of rotation which passes through the substrate.

10. A method as claimed in Claim 9, characterized in that the distance on a circular substrate surface between the axis of rotation which passes through the substrate and the average point of impact of the drop or drops of the liquid material is between $1/10$ and $1/3$ of the diameter of the substrate surface.

11. A method as claimed in Claim 10, characterized in that the said distance is between $1/5$ and $1/4$ of the diameter of the substrate surface.

12. A method as claimed in any of the preceding Claims, characterized in that the liquid material consists of the solid material in molten form.

13. A method as claimed in any of the preceding Claims, characterized in that the solid material is a crystalline material.

14. A method as claimed in Claim 13, characterized in that the solid material is a semiconductor material.

15. A method as claimed in Claim 14, characterized in that the semiconductor material is silicon.

16. A semiconductor device, in particular a solar cell, having a body of semi-

conductor material obtained by using a method as claimed in Claim 14 or 15.

- 5 17. A method of providing a layer of material on a flat substrate surface, substantially as herein described with reference to Fig. 1 or Fig. 2 of the accompanying drawings.

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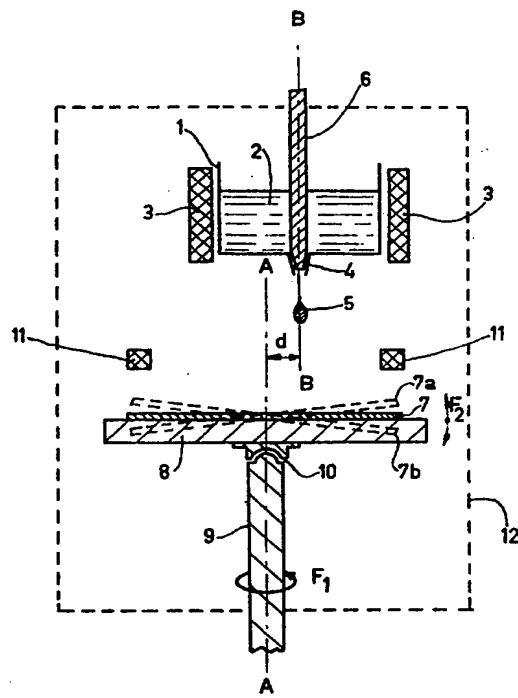


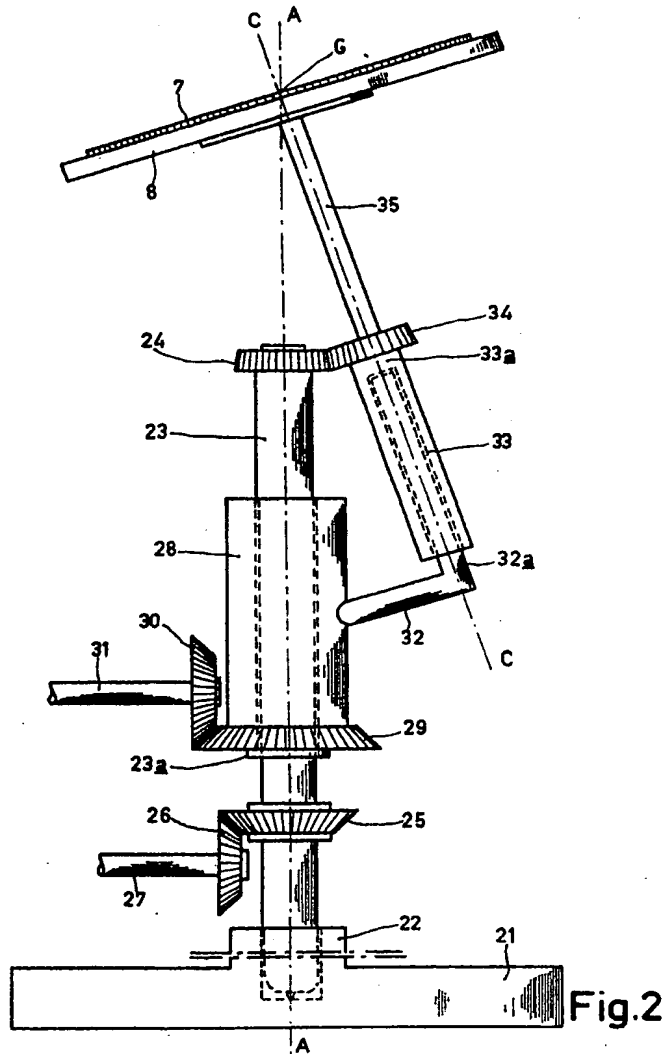
Fig.1

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COMPLETE SPECIFICATION

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